METHOD 9058

DETERMINATION OF PERCHLORATE USING ION CHROMATOGRAPHY WITH CHEMICAL SUPPRESSION CONDUCTIVITY DETECTION

1.0 SCOPE AND APPLICATION

1.1 This method describes the determination of the perchlorate anion in water by ion chromatography.

1.2 The applicable matrices are listed below.

1.2.1 This method has been found to perform adequately on water samples with conductivities up to 1000 µS/cm. Water samples with conductivities >1000 µS/cm have not been tested.

1.2.2 This method is potentially applicable to surface water, mixed domestic water, and industrial wastewaters.

1.3 A method detection limit (MDL) for perchlorate in reagent water has been calculated to be 0.7 µg/L (pooled data, see Table 1). The MDL for a specific matrix will differ from that listed, depending upon the nature of the sample. When MDLs are needed for a specific project, matrix-specific MDL studies may provide data users a more reliable estimate of method detection capabilities.

1.4 The linear calibration range for perchlorate is approximately 4 to 500 µg/L. Sample concentrations higher than the upper calibration limit should be diluted with reagent water to a concentration within the calibration range and reanalyzed.

1.5 Figure 1 provides example chromatograms for 4 µg/L of perchlorate added to reagent water and to a groundwater sample.

1.6 When this method is used to analyze unfamiliar samples, perchlorate identification should be supported by a demonstration of performance (see Sec. 9.2).

1.7 Users should determine or establish the data quality objectives (DQOs) prior to analysis. Users must demonstrate the ability to generate results with this method that meet or exceed the DQOs, using the procedures described in Sec. 9.0.

1.8 Analysts should consult the disclaimer statement at the front of the manual and the information in Chapter Two, Sec. 2.1, for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is not mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.9 This method is recommended for use by analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

2.0 SUMMARY OF THE METHOD

2.1 A fixed volume of sample is injected into an ion chromatographic system, where the perchlorate anion is separated from other interfering anions and quantified.

2.2 A large volume sample loop is used to detect perchlorate in the low ppb (µg/L) range without sample preconcentration.

2.3 To minimize hydrophobic interaction of the perchlorate anion with the anion exchange resin, *p*-cyanophenol is added to the eluent to deactivate the active sites on the AS-5 resin. Without column deactivation, the perchlorate peak elutes later, is broader (isocratic elution), and tails severely, thus resulting in poor peak detection as the concentration of perchlorate in the sample decreases.

3.0 DEFINITIONS

Refer to Chapter One and Chapter Three for applicable definitions.

4.0 INTERFERENCES

4.1 Interferences can be caused by substances with retention times that are similar to and overlap the anion of interest. High concentrations of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.

4.2 The large water dip, or negative peak, is due to the large aliquot of sample injected onto the column. The perchlorate anion is retained for a sufficient length of time in the column, however, and elutes free of interference from the water dip.

4.3 Due to the strength of the eluent, the majority of the anions in a water sample will elute soon after the water dip. Because of the large sample volume injected, the detector response from these anions may be very high, depending on the amount of dissolved solids present in the sample. With the longer retention time, the perchlorate anion elutes on the tail end of these early eluting anions and therefore, the detection and quantification of perchlorate is largely unaffected. Refer to Figure 1.

4.4 Method interferences may be caused by contaminants in the reagents, reagent water, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in ion chromatograms.

4.5 Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to the instrument, columns and flow systems.

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

5.3 The following chemicals have the potential to be highly toxic or hazardous. The MSDS for each chemical should be consulted.

5.3.1 Sodium hydroxide (Sec. 7.2).

5.3.2 Sulfuric acid (Sec. 7.3).

5.3.3 Potassium perchlorate (Sec. 7.4).

5.3.4 *p*-Cyanophenol (Sec. 7.2).

6.0 EQUIPMENT AND SUPPLIES

6.1 Analytical balance - capable of accurately weighing to the nearest 0.1 mg.

6.2 Ion chromatograph - an analytical system complete with ion chromatograph and all required accessories, including syringes, analytical columns, compressed gasses and detectors.

6.3 Sample loop - approximately 740 µL (12' x 0.02" I.D. tubing).

6.4 Anion guard column - Dionex IonPac AG5 , or equivalent.

6.5 Anion separator column - Dionex IonPac AS5 , or equivalent. This column produces the separation shown in Figure 1. An IonPac AS11 column and corresponding guard column (AG11) are also appropriate for the determination of perchlorate when using an eluent of 100 mM sodium hydroxide. This more hydrophilic column does not require *p*-cyanophenol in the eluent and can be used in conjunction with an anion suppressor regeneration system (ASRS) device operated at 300 mA in the external water mode. An ASRS is not recommended when using the eluent described in Sec. 7.2.

6.6 Anion suppressor device - a suppressor device with a high dynamic suppression capacity, such as Dionex AMMS-II suppressor or equivalent. The AMMS should be used with a regenerant solution of 0.035N H_2SO_4. A Dionex ASRS-ULTRA electrolytic suppressor, operated at 300 mA in the external water mode, can be used in conjunction with the AS11 column and an eluent of 100 mM NaOH. An ASRS is not recommended when the eluent contains *p*-cyanophenol.

6.7 Conductivity detector - Dionex CDM-II, or equivalent.

6.8 Chromatography data system - the data presented in this method were generated using the Dionex ACI-I computer interface and the Dionex AI-450 Data Chromatography Software. An equivalent data collection and chromatography processing system may also be used.

6.9 Sample bottles - polyethylene, 125-mL, or larger.

6.10 Volumetric flasks and pipettes, Class A - assorted sizes.

6.11 Filters and filter apparatus - 0.2- and 0.45-µm.

7.0 REAGENTS AND STANDARDS

7.1 Reagent water - ASTM Type I or deionized water, free of the anion of interest. The reagent water should not contain particles larger than 0.20 µm.

7.2 Eluent solution - 50% (w/w) sodium hydroxide (CASRN 1310-73-2) 120 mM, *p*-cyanophenol (CASRN 767-00-0) 2.0 mM. Dissolve 19.20 g of 50% (w/w) sodium hydroxide (NaOH) and 0.4765 g of *p*-cyanophenol (NCC_6H_4OH, 95%, Aldrich, or equivalent) in degassed reagent water and dilute to 2 L. The 50% (w/w) NaOH should be freshly prepared, with minimal contamination from dissolved CO_2 (carbonate formation). For the AS11 column (see Sec. 6.5), an eluent of 100 µm of sodium hydroxide is recommended. Dissolve 16 g of 50% w/w sodium hydroxide in degassed reagent water and dilute to 2 L.

7.3 Regenerant solution (micro-membrane suppressor) - sulfuric acid (CASRN 7664-93-9) 0.035N. Dilute 3.9 mL of reagent grade concentrated sulfuric acid (H_2SO_4) to 4 L with reagent water.

7.4 Stock standard perchlorate solutions, 1000 mg/L (1 mg/mL) - the stock standard solution is prepared from ACS reagent grade material. Dissolve 1.3931 g of potassium perchlorate ($KClO_4$, CASRN 7778-74-7) in reagent water and dilute to 1 L. Certified standards may also be purchased and used.

7.4.1 Calibration standards - prepare a 1000 mg/L perchlorate ($KClO_4$) stock solution for use in preparing the instrument calibration solutions, LCS and MSD solutions.

7.4.2 Instrument calibration verification (ICV) - prepare a 1000 mg/L perchlorate stock solution using a material source different from that of the calibration stock for use in preparing the ICV. The ICV is used to verify the accuracy of the instrument calibration.

7.4.3 The analyst should be aware of the purity of the potassium perchlorate used to prepare the stock standard. A weight correction must be made when the solid material is less than 99% pure.

7.5 Intermediate stock standard perchlorate solutions - prepare standard solutions at concentrations of 10, 1.0 and 0.10 mg/L, from the stock standard solutions.

NOTE: Stability of standards - The stock standard is stable for at least one month when stored at 4°C. The intermediate stock and dilute working standards should be prepared weekly.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to ensure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

8.2 Samples collected for perchlorate analysis should be stored at 4 ± 2°C from the time of collection until analysis. Samples should be analyzed within 28 days of collection.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for additional guidance on quality assurance protocols. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Initial demonstration of proficiency

9.2.1 The initial demonstration of proficiency is used to characterize instrument performance and laboratory performance prior to performing analyses by this method.

9.2.2 Linear calibration range (LCR) - The LCR must be determined initially and verified every six months, or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use a sufficient number of standards to insure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceed the initial values by ± 10%, then linearity must be reestablished. If any portion of the range is shown to be nonlinear, then sufficient standards must be used to clearly define the nonlinear portion.

9.2.3 Method detection limit (MDL) - The MDL should be established for the analyte using reagent water (blank), as described in Chapter One. MDLs should be determined periodically, when a new operator begins work, or whenever a significant change occurs in the background or instrument response.

9.3 Assessing laboratory performance

9.3.1 Method blank - The laboratory must analyze at least one method blank with each batch of samples. The method blank data are used to assess contamination from the laboratory environment and sample processing (e.g., pre-filtration). Values that exceed the MDL indicate that laboratory or reagent contamination should be suspected and corrective action must be taken before continuing the analysis. The method blank must be carried through the entire preparation and analysis scheme, including filtration.

9.3.2 Laboratory control sample (LCS) - For each batch of samples processed, at least one laboratory control sample must be carried throughout the entire sample preparation and analytical process, as described in Chapter One. The laboratory control samples should be spiked with each analyte of interest at the project-specific action level or lacking project-specific action levels, between the low and mid-level standards. Acceptance criteria should be set at a laboratory-derived limit developed through the use of historical analyses. In the absence of historical data, this limit should be set at ± 20% of the spiked

value. Acceptance limits derived from historical data must be no wider that ± 20%. If the laboratory control sample is not acceptable, then the laboratory control sample must be re-run once. If the results are still unacceptable, then all samples analyzed after the last acceptable laboratory control sample must be reprepared and reanalyzed. Refer to Chapter One for more information.

9.3.3 Instrument calibration verification (ICV) - For all determinations, the laboratory must analyze the ICV (a second source mid-range check standard) and a calibration blank immediately following daily calibration, after every tenth sample and at the end of the sample run. Analysis of the ICV solution and calibration blank immediately following calibration must verify that the instrument is within ± 10% of calibration. Subsequent analyses of the ICV solution must verify that the calibration is still within ± 10%. If the calibration cannot be verified within the specified limits, reanalyze the ICV solution. If the second analysis of the ICV solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable ICV solution must be reanalyzed. The analysis data of the calibration blank and ICV solution must be kept on file with the sample analyses data.

9.4 Assessing analyte recovery and data quality

9.4.1 Matrix spike (MS) - The laboratory must perform a matrix spike on a minimum of 10% of the routine samples. The MS aliquot must be a duplicate of the aliquot used for sample analysis. The spiked perchlorate concentration must be large enough to be detected above the original sample concentration and should not be less than five times the MDL. The added perchlorate concentration should be the same as that used in the LCS.

9.4.1.1 In a blind matrix spike, if the concentration of fortification is less than 25% of the background concentration of the matrix, the matrix recovery should not be calculated and a new MS should be prepared, analyzed, and reported.

9.4.1.2 Calculate the percent recovery for perchlorate, corrected for the concentration measured in the unfortified sample, and compare the value to the initial MS recovery range of 75-125%. The recovery limits should never exceed 75-125%. Percent recovery may be calculated using the following equation:

$$\%\ R = \frac{C_A - C}{A} \times 100$$

where: %R = percent recovery
C_A = fortified sample concentration
C = sample background concentration
A = concentration equivalent to analyte added to sample

9.4.1.3 When sufficient internal performance data become available (a minimum of 20 analyses), develop control limits from percent mean recovery (X) and the standard deviation (S) of the mean recovery.

9.4.1.4 If the recovery of the analyte falls outside the designated MS recovery range and the laboratory performance for that analyte is shown to be in

control (Sec. 9.3), the recovery problem encountered with the MS is judged to be either matrix or solution related, not system related.

9.4.2 Where reference materials are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to acceptably perform the method.

9.4.3 In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options, such as the use of an anion concentrator column, different columns and/or eluents, to improve the separation, quantification, or lower the cost of measurements. Each time such modifications to the method are made, the analyst must repeat the procedure in Sec. 9.2.

9.4.4 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The most productive practices will depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to monitor the precision of the sampling technique. When doubt exists over the identification of a peak in the chromatogram, confirmatory techniques, such as sample dilution and fortification, must be used. Whenever possible, the laboratory should perform analysis of quality control check samples and participate in relevant performance evaluation sample studies.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Establish ion chromatographic operating parameters equivalent to those indicated in Table 1, or as recommended by the instrument manufacturer.

10.2 Prepare a blank and calibration standards at a minimum of five concentrations by adding accurately measured volumes of one or more intermediate stock standards (Sec. 7.5) to a volumetric flask and diluting to volume with reagent water. The low calibration standard should be at or slightly above the laboratory's reporting limit. Perform a full instrument calibration on a monthly basis, or whenever a significant change in instrument response is observed or expected.

10.2.1 During this procedure, the perchlorate retention time must be recorded.

10.2.2 To confirm the linearity of the calibration curve, the predicted concentration for each calibration standard should be calculated by using the established linear regression curve and response from each standard concentration. If the predicted response for any standard varies from the expected response by more than ± 10%, perform corrective action.

10.3 The calibration curve must be verified by analyzing the ICV solution on each working day, or whenever the anion eluent is changed, and after every 10 samples. If the response or retention time for perchlorate varies from the expected values by more than ± 10%, then the test must be repeated, using fresh ICV solution. If the results are still more than ± 10%, then a new calibration curve must be prepared.

11.0 PROCEDURE

11.1 Table 1 summarizes the recommended operating conditions for the ion chromatograph. The table includes examples of the MDL and retention time that may be achieved with this method.

11.2 Check the system calibration (Sec. 10.3) and, if required, recalibrate as described in Sec. 10.0.

11.3 Analyze the samples along with appropriate QC samples.

11.4 The width of the retention time window used to make the perchlorate identification should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

11.5 If a sample concentration exceeds the calibration range, the sample must be diluted with reagent water to fall within the working range, and reanalyzed.

11.6 If the resulting chromatogram fails to produce adequate resolution, or if identification of the specific anion is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

NOTE: Retention time is inversely proportional to concentration. In some cases, this peak migration may produce poor resolution or identification.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Peak integration may be performed using either the peak height or peak area method, but must be uniform for all standards, samples, and QC samples.

12.2 Examine the chromatograms for perchlorate baselines set by the parameters used in the chromatography method. Correct any baseline improperly set by the method by modifying the integration parameters in the method. Save the corrected baseline to the raw data file.

12.3 Prepare the calibration curve by plotting the instrument response against the standard concentration. Compute the sample concentration (corrected for any sample dilution) by comparing the sample response with the standard curve.

12.4 Samples exceeding the highest standard should be diluted and reanalyzed.

13.0 METHOD PERFORMANCE

13.1 Table 1 contains an example of a single-laboratory MDL obtained under the conditions listed. These data are provided for illustrative purposes only.

13.2 Tables 2, 3, and 4 contain example single-laboratory accuracy and precision for perchlorate in reagent water and in groundwater obtained under the conditions listed. These data are provided for illustrative purposes only.

13.3 Tables 5, 6, and 7 contain example multi-laboratory accuracy and precision for perchlorate in reagent water and in groundwater obtained under the conditions listed. These data are provided for illustrative purposes only.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasiblely reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society, 1155 16th Street NW, Washington, DC 20036, (202) 872-4477.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, by complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Sec. 14.3.

16.0 REFERENCES

1. Record 269, Dionex Chromatography Database 4.2.0, Dionex Corp., Sunnyvale, CA, 94086.

2. U. S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Office of Research and Development, Cincinnati, OH, "Method 300.0, Revision 2.1, Determination of Inorganic Anions by Ion Chromatography," August 1993.

3. S. Chaudhuri, H. Okamoto, S. Pia, D. T. Tsui, "Inter-Agency Perchlorate Steering Committee Analytical Subcommittee Report."

4. California Department of Health Services, "Determination of Perchlorate by Ion Chromatography," Rev. 1.0, March 3, 1999.

17.0 TABLES, DIAGRAMS, FLOW CHARTS AND VALIDATION DATA

The pages to follow contain Tables 1 through 7, Figure 1, and a flow diagram of the method procedure.

TABLE 1

SUGGESTED CHROMATOGRAPHIC CONDITIONS
AND AN EXAMPLE METHOD DETECTION LIMIT IN REAGENT WATER

Perchlorate Spike Conc. (µg/L)	No. of Replicates	Mean Perchlorate Recovery (µg/L)	Standard Deviation (µg/L)	Calculated MDL (µg/L)
1.0	14	0.87	0.11	0.6
2.5	16	2.3	0.12	0.8
4.0	16	3.9	0.11	0.7

Pooled MDL (*df* = 43) 0.7 µg/L
Retention Time 7.4 min
Perchlorate peak height response for 4.0 µg/L ≈ 0.04 µS

Data taken from Reference 1 and are provided for illustrative purposes only.

Chromatographic conditions used to produce the data in this method:

Instrument	Dionex 4500 Ion Chromatograph with Autosampler
Detector	Dionex CDM-2
Ion Suppressor	Dionex AMMS-II
Columns	Dionex IonPac AG5 Guard column
	Dionex IonPac AS5 Analytical
Column Temperature	Ambient
Injector Loop	740 µL (approximate volume)
Eluent	120 mM NaOH + 2.0 mM *p*-Cyanophenol
Eluent Flow rate	1.0 mL/min
Regenerant	35 mN H_2SO_4
Regenerant flow rate	10 mL/min
Conductivity Detector Background Reading	<12 µS

TABLE 2

EXAMPLE SINGLE-OPERATOR ACCURACY AND PRECISION FOR PERCHLORATE STANDARD SOLUTIONS

Sample Type	Known Conc. (µg/L)	Number of Replicates	Mean Recovery (µg/L)	Mean Recovery (%)	SD (µg/L)	RSD (%)
ICV Standard	5.0	48	4.9	98	0.35	7.1
	100	47	100	100	4.2	4.2
Laboratory Control Sample	4.0	16	4.0	100	0.31	7.8
	100	4	100	100	2.8	2.8
Spiked Blank	4.0	22	3.9	98	0.33	8.5

All sample types were prepared in reagent water.

Data taken from Reference 4 and are provided for illustrative purposes only.

TABLE 3

EXAMPLE SINGLE-OPERATOR ACCURACY AND PRECISION
FOR PERCHLORATE SPIKED INTO GROUNDWATER SAMPLES

Sample Type	Spike Conc. (µg/L)	Number of Spiked Pairs	Duplicate Spike Mean Recovery (µg/L)	Duplicate Spike Mean Recovery (%)	Mean RPD (%)	SD of Mean RPD (%)
Matrix Spike/ Matrix Spike Duplicate	4.0	20	3.8	95	2.1	0.02

All MS/MSD pairs were prepared in groundwater.

Data taken from Reference 4 and are provided for illustrative purposes only.

TABLE 4

EXAMPLE SINGLE-OPERATOR PRECISION FOR PERCHLORATE SAMPLE REPLICATES

Sample Type	Number of Replicate Pairs	Mean RPD (%)	SD of Mean RPD (%)
Sample/Sample Duplicate	14	1.4	0.02

All samples consisted of groundwater with perchlorate concentration $\geq$ 4.0 µg/L.

Data taken from Reference 4 and are provided for illustrative purposes only.

TABLE 5

EXAMPLE MULTI-LABORATORY DATA FOR AS-11 AND AS-5 COLUMNS

Sample ID	Columns	No. of Labs	No. of Samples	Mean of All Samples (ppb)	Standard Deviation (ppb)
C2T1	AS-11	12	36	5.75	0.72
	AS-5	4	12	5.96	0.62
	Combined data	17	51	5.81	0.69
C2T2	AS-11	13	39	5.75	0.70
	AS-5	4	12	5.96	0.51
	Combined data	18	54	5.82	0.67
C2T3	AS-11	11	33	6.04	0.77
	AS-5	3	9	5.90	0.38
	Combined data	15	45	6.06	0.72
C3T1	AS-11	11	33	17.9	1.3
	AS-5	5	15	18.2	1.2
	Combined data	17	51	18.0	1.2
C3T2	AS-11	12	36	17.8	1.4
	AS-5	4	12	17.6	1.2
	Combined data	17	51	17.8	1.3
C3T3	AS-11	12	36	18.2	2.1
	AS-5	5	15	17.6	2.0
	Combined data	18	54	18.1	2.0
C4T1	AS-11	13	39	34.6	2.7
	AS-5	5	15	36.0	2.6
	Combined data	19	57	35.0	2.6
C4T2	AS-11	11	33	36.0	2.2
	AS-5	5	15	36.7	2.9
	Combined data	17	51	36.2	2.4
C4T3	AS-11	12	36	35.6	2.7
	AS-5	5	15	35.5	3.2
	Combined data	18	54	35.6	2.7
STO	AS-11	26	78	51.00	1.92
	AS-5	15	45	51.61	2.40
	Combined data	44	132	51.18	2.07

Data taken from Reference 3 and are provided for illustrative purposes only.

TABLE 6

EXAMPLE MULTI-LABORATORY ACCURACY AND BIAS FOR AS-11 AND AS-5 COLUMNS

	Recovery (%)		Bias (%)		CV (%)	
Sample ID	AS-11	AS-5	AS-11	AS-5	AS-11	AS-5
C2T1	95.9	99.3	-4.1	-0.7	11.5	10.7
C2T2	95.8	99.3	-4.2	-0.7	10.3	8.6
C2T3	95.2	99.4	-4.8	-0.6	12.1	5.4
C3T1	99.2	101.0	-0.8	1.0	6.7	5.5
C3T2	97.7	101.3	-2.3	1.3	8.0	4.4
C3T3	99.7	98.4	-0.3	-1.6	9.5	3.4
C4T1	96.2	99.3	-3.8	-0.7	7.6	4.1
C4T2	97.3	101.5	-2.7	1.5	6.0	6.6
C4T3	97.5	98.7	-2.5	-1.3	7.4	5.9
ST0	101.4	101.3	1.4	1.3	3.3	3.1
Mean	97.6	99.9	-2.4	-0.1	8.2	5.8
Std. Deviation	8.2	5.8	—	—	—	—

Data taken from Reference 3 and are provided for illustrative purposes only.

TABLE 7

EXAMPLE DATA FROM IPSC COLLABORATIVE STUDY SAMPLES

Sample ID	Total Dissolved Solids (ppm)	Expected Perchlorate (ppb)	Measured Perchlorate (ppb)	Standard Deviation (ppb)	Number of Samples
C1/T1-3	-	0	0	-	10
C2T1	72	5.8	6.50	0.75	5
C2T2	144	5.8	6.19	0.81	5
C2T3	288	5.8	6.74	0.87	5
C3T1	72	17.9	17.8	1.0	5
C3T2	144	17.9	18.1	~1.7	5
C3T3	288	17.9	18.4	1.9	5
C4T1	72	36	34.1	1.9	5
C4T2	144	36	37.0	4.6	5
C4T3	288	36	35.9	2.9	5
S/TO	-	50	51.4	3.8	10

C1/T1-3 is a reagent water blank.

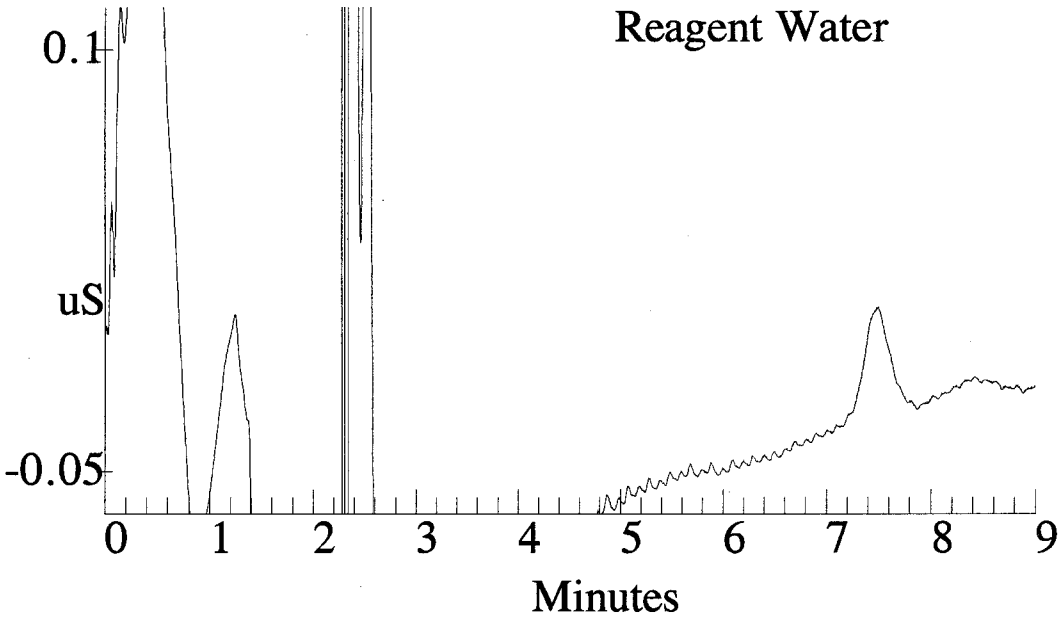
S/TO is a reagent water standard.

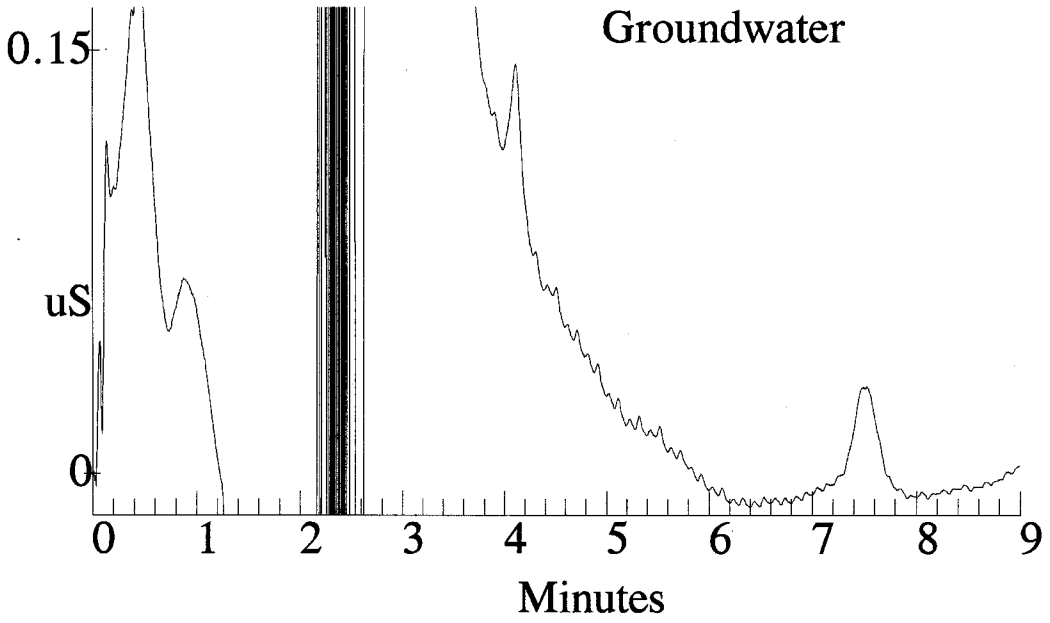
C2xx-C4xx are spiked well water samples.

Data taken from Reference 3 and are provided for illustrative purposes only.

FIGURE 1

EXAMPLE CHROMATOGRAMS OF REAGENT WATER AND GROUNDWATER SAMPLES CONTAINING 4 µg/L OF PERCHLORATE





DETERMINATION OF PERCHLORATE USING ION CHROMATOGRAPHY WITH CHEMICAL SUPPRESSION CONDUCTIVITY DETECTION